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PROJECT LEADER: J. I. Seeman
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I. INORGANICS AS NOVEL TOBACCO MATERIALS ADDITIVES (Fournier, Paine, Podraza, Seeman)

A. Objective: To develop inorganic materials for novel applications for reduced sidestream and enhanced subjectives in cigarettes and for required properties in novel smoking materials.

B. Results and Plans: A series of CaX_2 (X = Cl, OAc) + M_2CO_3 (M = Na, K) reactions were performed, some in the presence of a dispersing agent (glycerol, diethylene glycol) to form $CaCO_3$ gel/particles. Gel stability was quantified by time for phase separation (immediate to 60 min). The particle sizes were determined by SEM and ranged from 0.01-6.00 microns with different morphologies.

Vanillin was found to be incorporated into the layers of hydrotalcite while eugenol, raspberry ketone, and isovanillin were not. Tube furnace pyrolysis of the hydrotalcite-vanillin product gave low yields of veratraldehyde, presumably due to an unprecidented transmethylation reaction within the layers of the inorganic moiety. A variety of magnesium, potassium and calcium double carbonates were prepared. A large scale preparation of calcium ethoxide was prepared per request from A. Kallianos.

The catalytic effects of iron oxides are being examined. Three samples with iron concentrations of 0%, 2% and 5% in alumina were prepared by codecomposition of the nitrates at 425°C. After further pulverization these samples will be submitted for papermaking as well as other standard characterization techniques. The alumina/iron oxide sample sent for x-ray diffraction indicated the sample to be amorphous. A sample has been submitted to Physical Research for confirmation. Microscopy of this same sample showed iron present in all crystallites (0.02 - 0.05 μm) examined. Another sample of AlOOH/5%FeOOH has been prepared by adding the appropriate amount of iron nitrate to Union Carbide's boehmite, peptizing, and precipitating the solids with KOH.

II. REMOVAL OF NICOTINE FROM AQUEOUS TOBACCO PROCESSING FLUIDS (Howe, Secor, Seeman)

A. Objective: To develop techniques to remove selectively nicotine and other alkaloids from aqueous tobacco processing fluids.

B. Results and Plans: We have continued to examine CR-2824, a tris(2-ethylhexyl)-derivative of tartaric acid as the liquid ion exchanger (LIX) for the membrane-based extraction process. This LIX results in excellent and reproducible nicotine extraction efficiencies. A large scale preparation of CR-2824 was completed.

Careful studies have shown that some of this LIX is transferred to the Tobacco Extract Solution (TES), even though it is nearly insoluble in water. We estimate that ca. 0.96 g of this LIX was transferred from the Organic Solvent (OS) to the TES during four 4-L runs (16 L of TES), or ca. 60 mg/L of TES. We conclude that it is the slight water solublity of the nicotine-LIX complex which drives the transfer, and that it may be possible to decrease the overall transfer to TES by back-transfer to OS by running each extraction to a greater percentage completion. This hypothesis will be tested shortly.

III. CHEMICAL PHYSICS STUDIES OF TOBACCO CONSTITUENTS (Secor, Seeman)

A. Objective: To obtain structural information about important tobacco constituents/flavorants; to develop information on cluster formation and chemical reactions in clusters.

B. Results and Plans: A series of six benzylamines were prepared and will soon be sent to E. Bernstein. These are being investigated as analogs of nicotine and nornicotine. We are in the process of examining the clusters of these benzylamines with CO_2 and H_2O, to add experimental information regarding the relative extractability of the tobacco alkaloids in the ART process. Final reports are being written on benzyl alcohols, styrenes, and alkylpyrazines.

IV. FLAVOR/ODOR CHEMISTRY (Podraza)

A. Objective: To prepare new substances for flavor/odor evaluation.

B. Results and Plans: The natural product 3,6-dimethyl-3a,4,5,7a-tetrahydro-2(3H)-benzofuranone (CR-2818) was prepared in a two step process. A 0.5 gram sample of CR-2818 was delivered to Tom Gannon in Flavor Development for subjective evaluation. 6-n-Butylhexahydro-2(3H)-benzofuranone was prepared as a mixture of diastereomers (trans/cis ratio, 10/90).